

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:

Fred S. Cannon et al.

Title:

METHOD OF HEAT TREATING ACTIVATED CARBON TO

ENHANCE ADSORPTION OF TASTE AND ODOR COMPOUNDS

AND TOTAL ORGANIC CARBONS

Serial No:

10/085,469

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### **Mail Stop Amendment**

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

# **DECLARATION UNDER 37 C.F.R. 1.132**

#### Dear Sir:

- 1. I, Fred S. Cannon, hereby declare and state the following:
- 2. I am a citizen of United States of America. I reside at 107 Ridgewood Circle, State College, PA 16803.
- 3. I have been continuously employed by The Pennsylvania State University from 1993 to the present. Currently, I am an Associate Professor of Civil and

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Environmental Engineering at the Civil and Environmental Engineering Department of The Pennsylvania State University.

- 4. I am a co-inventor of the Formal U.S. Patent Application Serial No. 10/085,469, filed on February 28, 2002, which claims priority from Provisional U.S. Patent Application Serial No. 60/272,091, filed on February 28, 2001.
- 5. I am also a co-inventor of the subject matter and experimental results described and presented herein including the table, herein after Table 1, which summarizes the results obtained and presented in this Declaration.
- 6. Table 1 relates to Pore Volume Distributions (cm<sup>3</sup>/g) for Representative Activated Carbons that are described in the above identified U.S. Patent Application Serial No. 10/085,469, which were prepared by the method according to the present invention of heat treating activated carbon to enhance adsorption of taste and odor compounds and total organic carbons.
- 7. Claims 1-8, 30 and 36-40 of the pending U.S. Patent Application Serial No. 10/085,469 have been rejected under 35 U.S.C. §102(b) as being anticipated by or. in the alternative, under 35 U.S.C. §103(a) as being obvious over Murakami et al. (U.S. Patent No. 6,865,068), herein after Murakami et al.
- 8. In claim 1, Murakami et al. claim a carbonaceous material defined as follows:

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"1. A carbonaceous material which has a total pore volume of from 0.5 to 1.5 cm³/g per unit mass, a volume of micropores having diameters of from 10 to 20 A of from 10 to 45 % based on the total pore volume, a volume of mesopores having diameters of from 20 to 200 A of from 35 to 65 % based on the total pore volume, a volume of macropores having diameters exceeding 200 A of not more than 15 % based on the total pore volume, and a specific surface area of from 1,000 to 2,500 m²/g."

In contrast, the claimed carbonaceous material in claim 1 of the pending U.S. Patent Application Serial No. 10/085,469 is defined as follows:

## "1. An activated carbon comprising:

a pore volume per gram of said activated carbon more than about 0.32 mL in the pore width range between about 4 to 63 angstroms; and

a pore volume per gram of said activated carbon more than about 0.21 mL in the pore width range between about 63 to 500 angstroms; provided that the pore volume per gram of said activated carbon in the pore width range of about 20 to 63 angstroms is at least about 25% of the total pore volume per gram of said activated carbon in said pore width range of 4 to 63 angstroms, as measured per the Argon Adsorption Density Functional Theory protocol and provided that said activated carbon exhibits a pH equal to or greater than 9.9, when immersed as a slurry in nitrogen-purged deionized distilled water, while the slurry contains about 10% by weight of activated carbon, as measured per the Slurry pH protocol."

The volume distribution measurements in the pending U.S. Patent Application Serial No. 10/085,469 are based on argon adsorption method. This method offers data on the micropores and mesopores, but it inherently cannot provide information on the macropores. In order to characterize the macropores, we have conducted mercury porosimetry analyses on several of these activated carbon samples that we have kept in our laboratory. Macropores, according to IUPAC definition, include all pores up to the

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size of spaces between one grain and another. Thus, herein, we have identified macropores to include those with diameters from 200 A to 20,000 A. The lower limit for macropores recited in claim 1 of Murakami et al. is 200 A.

The Mercury Porosimetry analyses were conducted at The Pennsylvania State University with Carlo Erba Pascal Model 140 and 440 Mercury Porosimeters, using the 140 model for lower pressures and the 440 model for higher pressures. The protocol was in accordance with the manufacturer's recommendations. Mercury pressures progressed from ambient up to 400 MPa, with >1000 data points of progressively increasing pressure. As pressure increased, the pore width that mercury could penetrate decreased. The pressure of 0.725 MPa corresponded to pore diameters of 20,000 A, while the 29.4 MPa pressure corresponded to pore diameters of 500 A. This is in accordance with the manufacturer's porosimeter software package which employs the Washburn equation:

 $pr = -2y \cos \theta$ 

wherein:

r = pore radius;

y = mercury surface tension;

 $\theta$  = contact angle; and

p = absolute applied pressure.

Activated carbon sample mass was 0.11 to 0.25 grams, and these were placed in a 465 mm<sup>3</sup> dilatometer. The mercury contact angle was taken to be 140°C, mercury surface tension was 480 Dynes/cm, mercury density was 13.54 g/cm<sup>3</sup>, and mercury temperature was 21°C. Duplicates were conducted for several samples, and duplicates were on average within 15% of one another.

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The argon adsorption data, which are included in the pending U.S. Patent Application Serial No. 10/085,469, was used here to depict pores in the < 500 A and in the 200 to 500 A diameter range.

The mercury porosimetry results, coupled with the argon adsorption results from the pending U.S. Patent Application Serial No. 10/085,469, are as shown in Table 1.

Table 1: Pore Volume Distributions (cm<sup>3</sup>/g) for Representative Activated Carbons that were included in the pending U.S. Patent Application Serial No. 10/085,469

Activated Carbon	Argon Adsorp- tion Pore Volume <500 A diameter	Argon Adsorption Pore Volume, 200-500 A diameter	Mercury Porosimetry Pore Volume 500 to 20,000 A diameter	Total Pore Volume <20,000A diameter	Pore Volume 200 to 20,000 A diameter	%volume of macropores having diameters exceeding 200 A
CH <sub>4</sub> /H <sub>2</sub> O (1000)-1	0.71	0.04	0.29	1.00	0.33	33
H <sub>2</sub> O (1000)	0.67	0.09	0.35	1.02	0.44	43
Pilot A	0.61	0.11	0.39	1.00	0.50	50
Pilot B	0.64	0.11	0.40	1.04	0.51	49
Pilot C	0.94	0.22	0.44	1.38	0.66	48
Commercial Lignite A	0.46	0.08	0.33	0.79	0.41	52

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- 9. It can be seen from Table 1 above, that for the Activated carbons of the Invention, the % volume of macropores having diameters exceeding 200 A ranged from 33 to 50 %, and this was considerably higher than the 15% maximum claimed by Murakami et al. Thus, the activated carbons described in the pending U.S. Patent Application Serial No. 10/085,469 are entirely different than those described by Murakami et al. Further, the activated carbons described in the pending U.S. Patent Application Serial No. 10/085,469 are neither taught nor suggested by Murakami et al.
- 10. I hereby declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true. I further declare that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Date: 12/8/2005

By:

Fred S. Cannon